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A multivariate statistic approach to detect process imbalance in lab scale continuously stirred tank anaerobic reactors submitted to inadequate organic loading rate

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INTRODUCTION

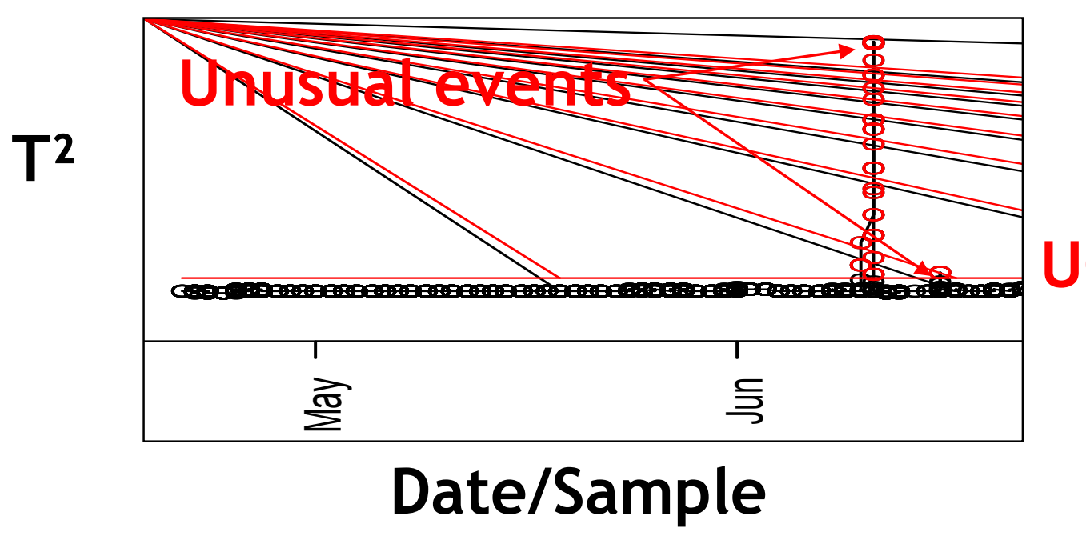
During recent decades, anaerobic digestion (AD) of organic substrates has become one of the most mature technologies to produce renewable energy from wet biomass. Biogas production using AD process implies to manage a complex microbial flora that is highly sensitive to variations of the ecological conditions existing in the reactors. The main biological dysfunctions are nowadays better understood and are for the most part related to inappropriate feeding of the digesters. So far various methods have been developed to monitor the process but none seems to be ideal. These methods usually consist in measuring a set of variables judged to be characteristic of the process status (i.e. pH of the liquid phase, CH_4/CO_2 ratio of the biogas,…) and interpreting the collected data for each parameter individually. However, since these variables reflect the conditions of the reactor anaerobic microbial community, it appears very probable that they present a certain degree of correlation. An efficient tool for AD process monitoring should therefore benefit from the integration of information about the way the measured parameters interact when the process is in control. A way to satisfy this condition is to monitor the reactors using multivariate statistic methods as an alternative to usual univariate approaches. Control charts built with Hotelling T² statistic are well adapted to this kind of application. Those charts are becoming more and more popular to monitor continuous processes were the quality of the final product is related to several dependant variables (Adam et al., 2012).

AIMS of the STUDY

Assessing the potential of Hotelling's multivariate control charts built from variables reputed as individual AD process indicators to detect dysfunction of anaerobic reactors submitted to inadequate feeding conditions.

Hotelling's control charts?

- The <u>most applied chart</u> in multivariate process control.
- **Phase I**: Data set built with measurements performed during a period where the process is supposed to be "in control". It authorizes to <u>compute 2 parameters that characterize this in-control situation</u> : the covariance matrix (*S*) and the mean vector (*Xmv*).
- **Phase II**: Each collected data sample is used to compute a unique statistical parameter (*T*²) that compare the current sample data structure with Phase I *Xmv* and *S*. T² value are compared to an upper control limit (UCL) to <u>identify out-of-control situations</u> :



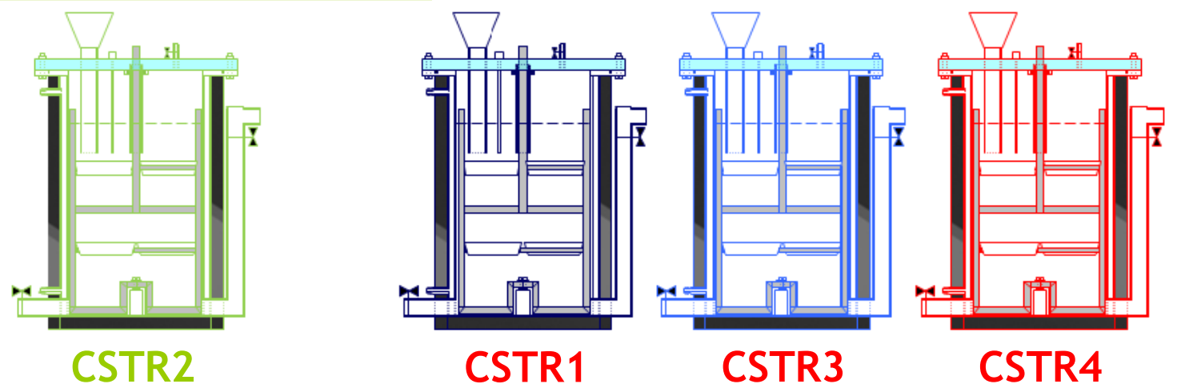
MATERIAL AND METHODS

Overfeeding campaigns with lab scale continuous reactors ⟷ **Continuous data collection** → **Analysis of the data sets with Hotelling's control charts**

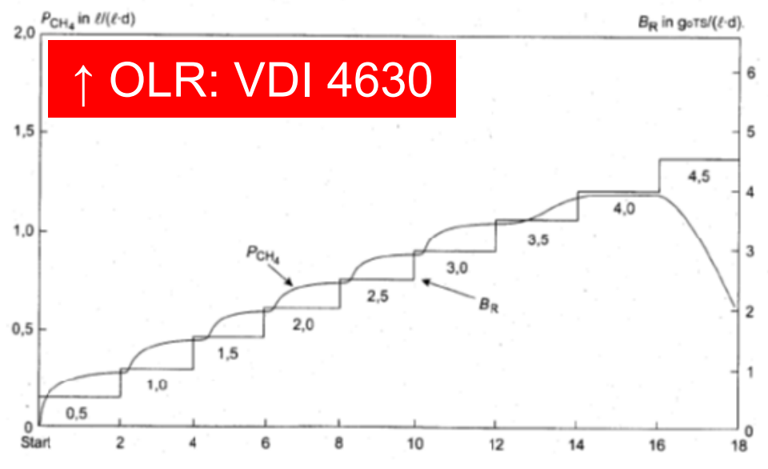
4 Continuously Stirred Tank Reactors (CSTR) 100l Mesophilic temperature range (37°C)

1 control reactor - cautious organic loading rate (OLR)

3 overfed reactors



- **<u>Inoculum:</u>** sludge from the anaerobic digester of a waste water treatment plant (mesophilic conditions).
- **<u>Substrate:</u>** dried sugar beet pulps.

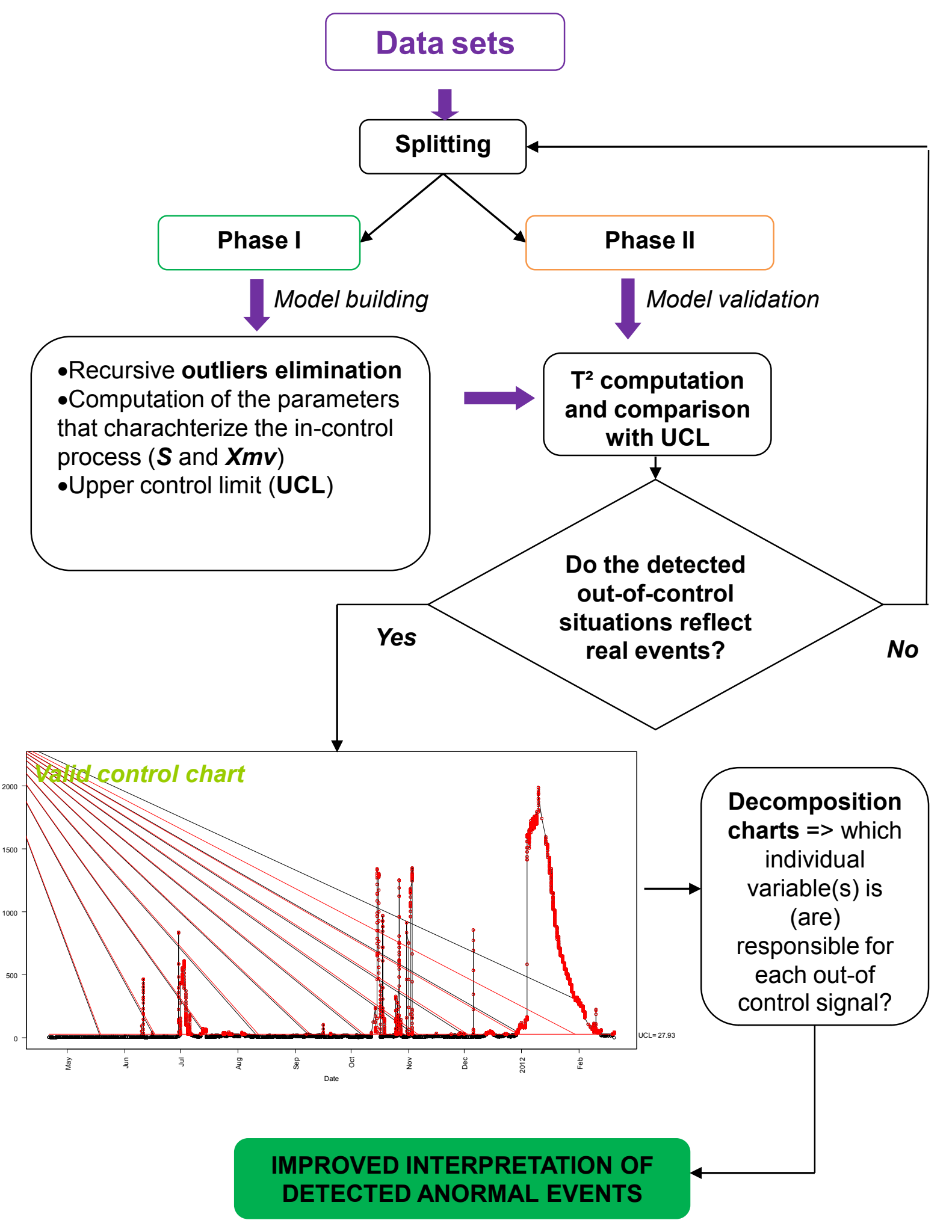


In the biogas :

Parameter	Units	Measurement method	Measurement Frequency
Biogas specific production	l/h	drum-type wet gas meter	h^{-1}
CH_4 concentration	% (Volume)	Non Dispersive Infra-Red Sensor (NDIR)	$(2h)^{-1}$
CO_2 concentration	% (Volume)	NDIR	$(2h)^{-1}$
H_2 concentration	ppm (Volume)	Metal Oxyde Semi-conductor (MOS) with molecular sieve	$(2h)^{-1}$
H_2S concentration	ppm (Volume)	Electro-Chemical Sensor (ECS)	$(2h)^{-1}$

In the digestate :

Parameter	Measurement method	Measurement Frequency
pH	Saturated calomel electrode	d^{-1}
Total solids (TS)	Gravimetry (VDI 4630)	w^{-1}
Volatile solids (VS)	Gravimetry (VDI 4630)	w^{-1}
Total alkalinity (TA)	Volumetric (BiogasPro, Germany)	w^{-1}
Ammoniac Nitrogen (NH_4-N)	Volumetric (BiogasPro, Germany)	w^{-1}



Data sets

Splitting

Phase I — *Model building*

- Recursive **outliers elimination**
- Computation of the parameters that charachterize the in-control process (***S*** and ***Xmv***)
- Upper control limit (**UCL**)

Phase II — *Model validation*

T² computation and comparison with UCL

Do the detected out-of-control situations reflect real events? ***Yes*** / ***No***

Decomposition charts => which individual variable(s) is (are) responsible for each out-of control signal?

IMPROVED INTERPRETATION OF DETECTED ANORMAL EVENTS

- Control charts were built using « MSQC » R package v1.0.1 (E. Santos-Fernandez)
- Decompositon chart were computed using « MYT » method (Mason et al., 1995)

RESULTS and DISCUSSION

1) Selection and optimization of the phase I data set (model building)

Phase I (T² after outliers cleaning) **Phase II (CSTR1)**

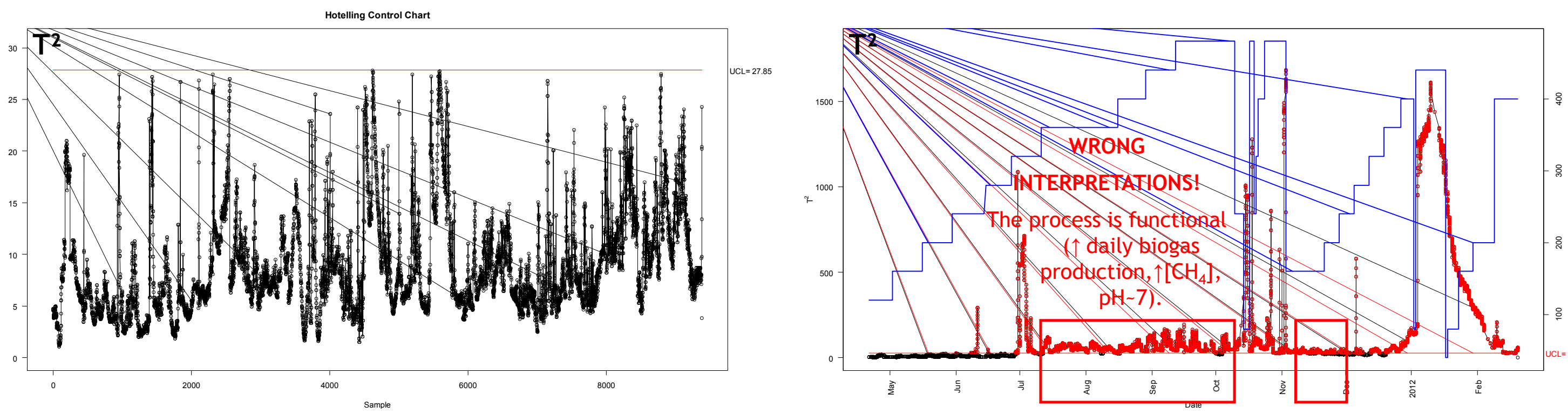


Fig. 1. All the measured parameters are used for model building except pH. Phase I is performed with a dataset issued from the cautiously fed ***control reactor*** (CSTR2). In phase II, an excessive number of samples are interpreted as out-of-control situations even though the digestion process is not perturbed.

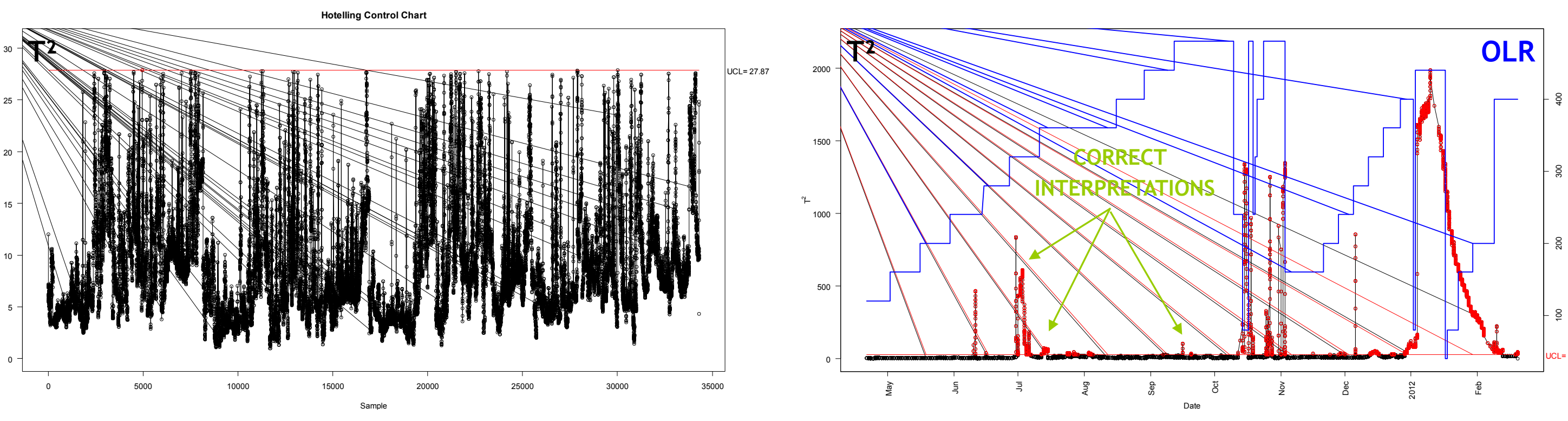


Fig. 2. All the measured parameters are used for model building except pH. Phase I is performed with a dataset combining ***mixed data from the 4 reactors***. In phase II, the detected unusual events can be related to real process dysfunction or real failures of the measurement system (cf. 2).

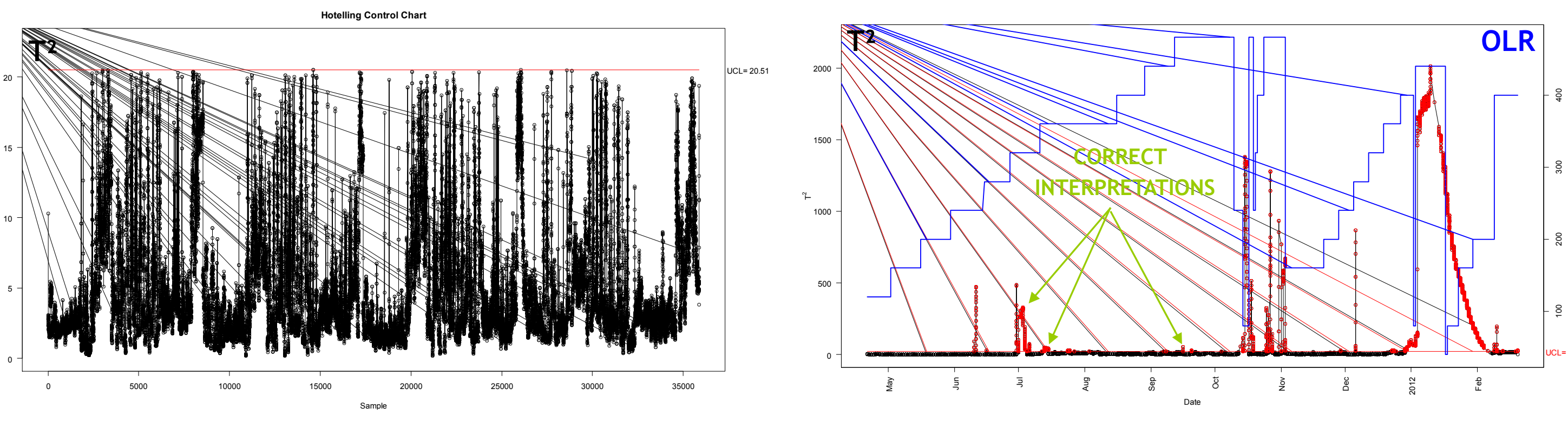
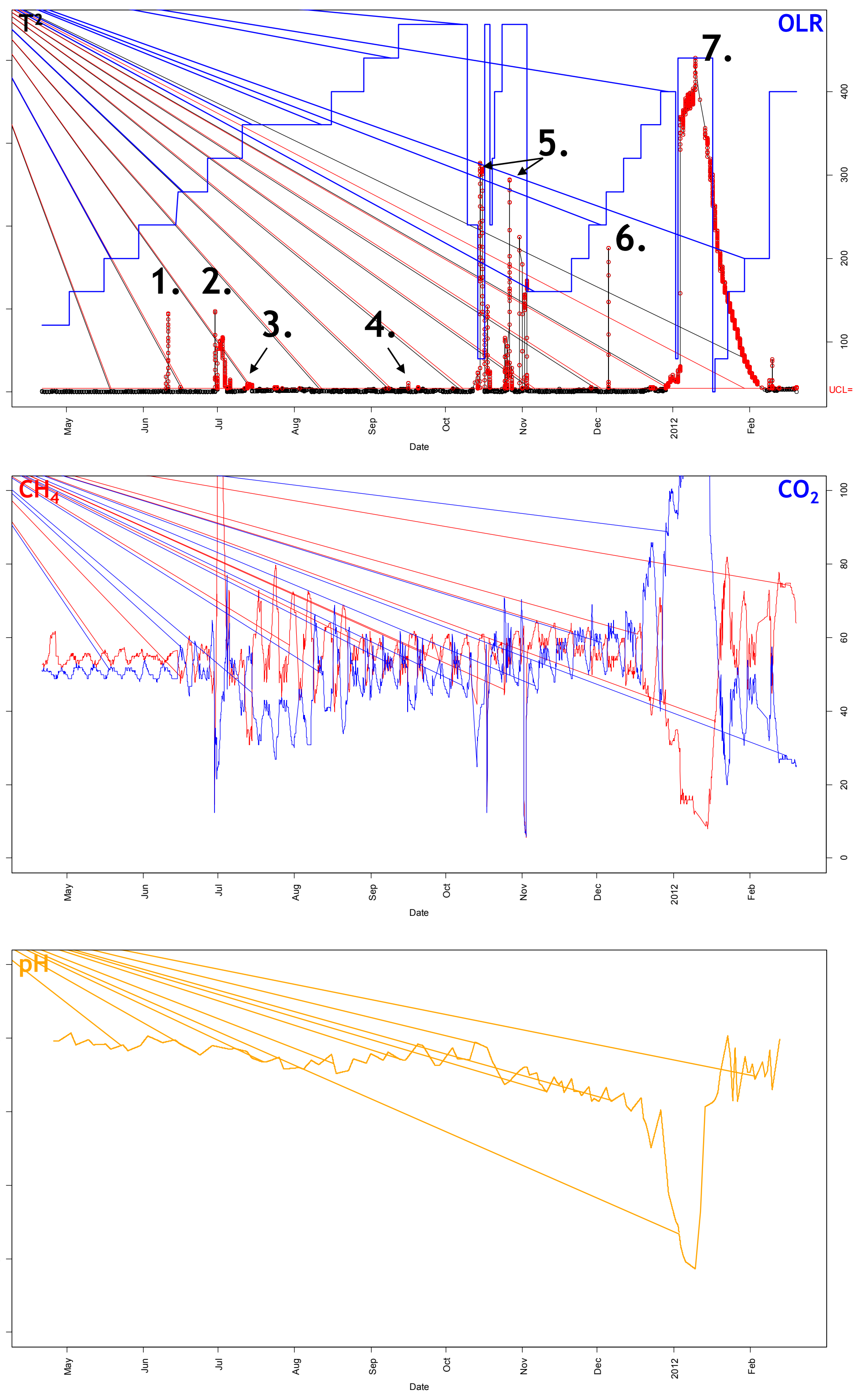


Fig. 3. Phase I is performed with a dataset combining mixed data from the 4 reactors but ***only the parameters measured in the gas phase are used***. Phase II still provide correct interpretation of the unusual events.

2) Process control for 1 overfed reactor (CSTR1)

NB : data set used for phase I was described Fig. 3. *pH of liquid phase is NOT included in the data sets.*



- **Event 1 and 6:** A stirrer malfunction occurred causing an important hydrogen production. Decomposition showed that hydrogen concentration in the biogas was the parameter responsible for the detected out-of-control situation (Fig. 4).
- **Event 2:** An important foam generation caused a failure of the gas sensors and required the disconnection of the biogas analyser for maintenance.
- **Event 3**: Drift of CH_4 and CO_2 sensors.
- **Event 4:** H_2S sensor was used to analyse a gas sample extremely concentrated. The sensor stayed perturbed during some hours after the measurement.
- **Event 5:** Unexplained hydrogen production peaks.
- **Event 7:** The reactor reaches an acidosis situation. The first out-of-control samples are related to an unusual relationship between CH_4 and CO_2 concentrations. Highest T² values are related to increasing hydrogen concentration.

Combinations of variables					t2 decomp	ucl	p-value
CH_4					0,17	10,83	0,678
CO_2					0,06	10,83	0,813
H_2					374,20	> 10,83	0
Biogas sp. pr.					0,27	10,83	0,604
H_2S					0,21	10,83	0,645
CH_4	CO_2				0,18	13,83	0,837
CH_4	H_2				374,49	> 13,83	0
CH_4	Biogas sp. pr.				0,32	13,83	0,728
CH_4	H_2S				0,42	13,83	0,656
CO_2	H_2				378,67	> 13,83	0
CO_2	Biogas sp. pr.				0,28	13,83	0,757
CO_2	H_2S				0,22	13,83	0,799
H_2	Biogas sp. pr.				410,38	> 13,83	0
H_2	H_2S				384,72	> 13,83	0
Biogas sp. pr.	H_2S				0,59	13,83	0,556
CH_4	CO_2	H_2			386,62	> 16,28	0
CH_4	CO_2	Biogas sp. pr.			0,32	16,28	0,810
CH_4	CO_2	H_2S			0,55	16,28	0,650
CH_4	H_2	Biogas sp. pr.			422,83	> 16,28	0
CH_4	H_2	H_2S			385,40	> 16,28	0
CH_4	Biogas sp. pr.	H_2S			0,64	16,28	0,590
CO_2	H_2	Biogas sp. pr.			410,78	> 16,28	0
CO_2	H_2	H_2S			395,40	16,28	0
CO_2	Biogas sp. pr.	H_2S			0,60	16,28	0,616
H_2	Biogas sp. pr.	H_2S			416,16	> 16,28	0
CH_4	CO_2	H_2	Biogas sp. pr.		439,77	> 18,49	0
CH_4	CO_2	H_2	H_2S		422,20	> 18,49	0
CH_4	CO_2	Biogas sp. pr.	H_2S		0,79	18,49	0,533
CH_4	H_2	Biogas sp. pr.	H_2S		429,11	> 18,49	0
CO_2	H_2	Biogas sp. pr.	H_2S		418,73	> 18,49	0
CH_4	CO_2	H_2	Biogas sp. pr.	H_2S	468,64	> 20,54	0

Fig. 4. Decomposition chart for event 1. All combinations of variables including hydrogen are responsible for the alarm.

CONCLUSIONS

- Hotelling's T² control charts appear as an ***interesting tool for AD process monitoring***. The study showed that it was possible to quickly detect out-of-control situations (related to both biological dysfunctions ant measurement system failure) using only gas phase parameters to build the model.
- T² is a ***unique parameter*** => the charts are easy to read and the T² value can be easily used to trigger various events to inform the manager of the reactors (i.e. alarm signal).
- Using the data of a reactor that was cautiously and steadily fed to define the in-control situation of the process did NOT appear as the best option. It was shown as more efficient to ***add to the training data set the measurements from overfed reactors***. After cleaning of the outliers, the data set included more information on how the variables interact each others for a wider range of OLR values. Subsequent event detection was then more realistic.
- In this study, ***liquid phase measurements did not seem to provide important added-value*** to the process control tool, probably because of their low frequency of measurement compared to biogas parameters.

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